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Fracture Behavior of Micro-Sized Fe-3%Si Alloy Single Crystals*

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Abstract

Fracture tests have been performed for both millimeter-sized and micro-sized specimens prepared from a Fe-3 mass%Si alloy single crystal, and the size effects on fracture behavior have been considered. Notch plane was set to be (100), which is a cleavage plane of this material, and notch direction was set to be [010] for both type of specimens. For millimeter-sized specimens, cleavage fracture occurred during introducing a fatigue pre-crack. In contrast, the micro-sized specimens were fractured by ductile manner. This phenomenon is discussed based on the plastic zone at crack tip and the ligament size. The results obtained in this investigation provide important information for designing actual MEMS devices.

Key words: Fracture, Micro-Sized Specimens, Micro Mechanical Testing, Size Effect

1. Introduction

MEMS (Micro Electro Mechanical Systems) devices [(1), (2)] are expected to be widely applied to devices for information technology, biomedical and automotive systems, and are being developed to global scale as one of platform techniques of the next generation. In MEMS devices, three dimensional microstructures are fabricated by micromachining techniques which are similar to the fabrication techniques of LSI [(1)]. To date, materials such as single crystal Si, polysilicon and Si_3N_4 have been used for MEMS devices, because these materials are fitted to the fabrication process of LSI. However, metallic, ceramic and polymeric materials are also considered to be applied to MEMS to improve to the performance of devices.

Length scale of the components used in MEMS devices is in the order of micron, as they are prepared from thin films deposited on substrates. In order to develop reliable MEMS devices, it is important to know the mechanical properties of thin film materials used in MEMS devices [(3)-(7)]. In particular, it is essential to know the fracture properties of thin films, as even micro/nano sized defects in the components provide stress concentration.

In addition, it is important to investigate the size effects on fracture behavior of micro-sized materials for designing MEMS devices [(8) (9)]. In quasi-brittle materials with crack or notch, the transition from brittle to ductile fracture has been observed when the specimen size decreases. This transition has been considered to occur when the ligament size of crack is close to a characteristic length (l_{ch}) [(10)-(12)]. However, this phenomenon has not been investigated wide range of length scale, and the size effect on fracture behavior in the micrometer order has not been reported.

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In this investigation, an Fe-3 mass%Si alloy single crystal [13], of which fracture behavior of macro-sized material is well known, is employed, and the size effect on fracture behavior in millimeter-sized and micro-sized specimens, which prepared from same Fe-3 mass%Si alloy single crystal, was discussed.

2. Experimental Procedure

2.1 Materials

The material used was an Fe-3 mass%Si alloy single crystal. This material is known to fracture by cleavage in bulk size [13], and the characteristic length (l_{ch}) is several hundred μm order. If we prepare two types of specimens, the ligament sizes of which are larger than that of l_{ch} and smaller that l_{ch}, it is possible to investigate the transition of fracture behavior from brittle to ductile on the micrometer scale. Table 1 shows chemical composition of this material. The crystallographic orientations of this material were determined by a back reflection X-ray Laue method.

Table 1 Chemical composition of a Fe-3mass%Si alloy single crystal. (mass%)

Fe	C	Si	Mn	P (ppm)	S (ppm)	O (ppm)	N (ppm)	H (ppm)
Bal.	0.0102	3.270	0.01	100	300	160	120	7

2.2 Specimen Preparation

2.2.1 Millimeter-Sized Specimen

Millimeter-sized three point bending specimen with dimensions of 11.4 (L) x 2 (W) x 2 (B) mm^3 was prepared by diamond wheel cutter. Notch with a width of 0.8 mm was introduced by electro discharge machining. The a/W ratio of the notch was set to be 0.4, where a is crack depth and W is specimen width. A fatigue pre-crack was introduced under the conditions of a maximum cyclic load of 71 N and a stress ratio (R) of 0.1 at room temperature in air by servo hydraulic type mechanical testing machine (Shimadzu Servo Pulser EHF-EDII5). Notch plane was (100), which is cleavage plane of this material, and notch direction was [010]. The notch position was set to center of the specimen. Figure 1(a) shows the orientation and shape of millimeter-sized specimen.

2.2.2 Micro-Sized Specimen

Micro-sized specimens were prepared by the following procedure. Thin foils with a thickness of 10 μm were prepared firstly by mechanical and chemical polishing. Micro-sized cantilever beam specimens with dimensions of 50 (L) x 10 (W) x 10 (B) μm^3 were fabricated by focused ion beam (FIB) machining. A notch with a depth of ~5 μm (width of notch = 0.25 μm) were introduced by focused ion beam machining, and a fatigue pre-crack was also introduced ahead of the notch under conditions of maximum cyclic load of 9~13 mN and stress ratio (R) of 0.1 at room temperature in air. The notch position was set to be 10 μm from the fixed end of the specimen, and the loading position was set to be 40μm from the fixed end of the specimen. Notch plane and notch direction were (100) [010], which are same as for millimeter-sized specimens. Figure 1(b) shows orientation and shape of micro-sized specimen, and Figure 2 shows a scanning electron micrograph of micro-sized Fe-3 mass%Si alloy single crystal specimen prepared by these procedures.

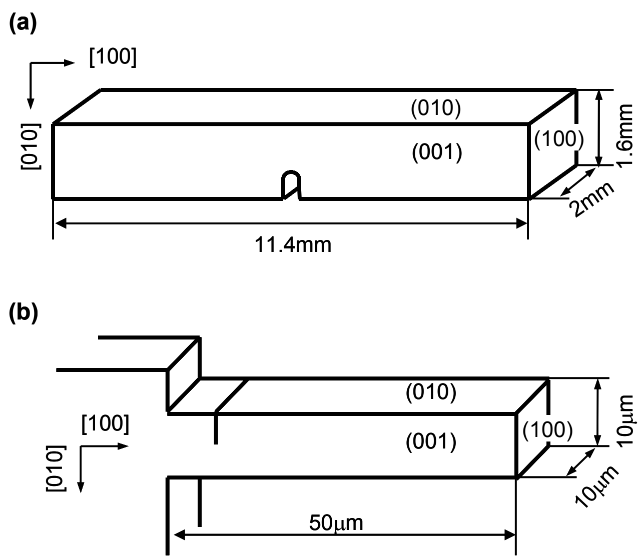

Fig. 1 Orientation and shape of specimens.

(a) millimeter-sized specimen. (b) micro-sized specimen.

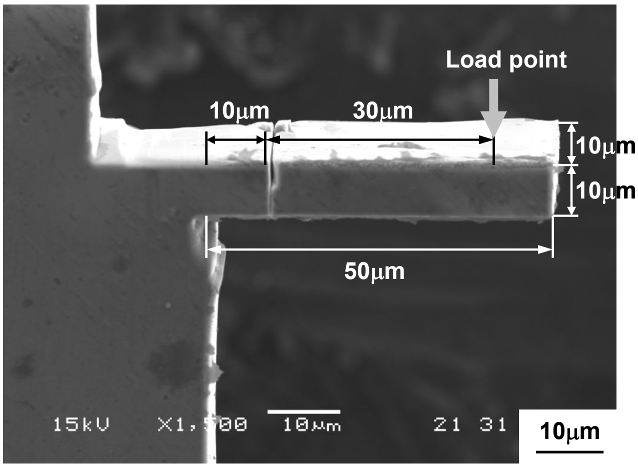

Fig. 2 Scanning electron micrograph of micro-sized Fe-3 mass%Si alloy single crystal specimen.

2.2 Fracture Testing

Fracture tests of millimeter-sized specimen were carried out at room temperature in air by the servo hydraulic type mechanical testing machine. Side surface and fracture surface of millimeter-sized specimen were observed by a scanning electron microscopy (JEOL JSM-5600) after fracture testing.

Fracture tests of micro-sized specimen were performed at room temperature in air using a mechanical testing machine developed by our group. Figure 3 shows a block diagram of the mechanical testing machine for micro-sized specimens. A micro-sized specimen was fixed on a special specimen holder and was set on a precision X-Y stage with a translational resolution of 0.05 μm. Actuator, which applies load on the specimen, used was a piezoelectric type device. A load cell was attached to the actuator, and a metallic rod was connected to them. A spherical diamond tip with a radius of 5 μm was attached to the other end of the rod, and the load is applied to the specimen through the diamond tip. Displacement is measured by a capacitive displacement sensor, which is integrated in the actuator, and feedback control was performed using the displacement signal.

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Displacement resolution is 0.2 nm. Strain gauge type load cell with a maximum load capacity of 200 mN and a load resolution of 20 μN was used. Mechanical testing machine was set on a vibration isolating table to remove the effect of vibration. Side surface and fracture surface of micro-sized specimen were also observed by the scanning electron microscope after fracture testing.

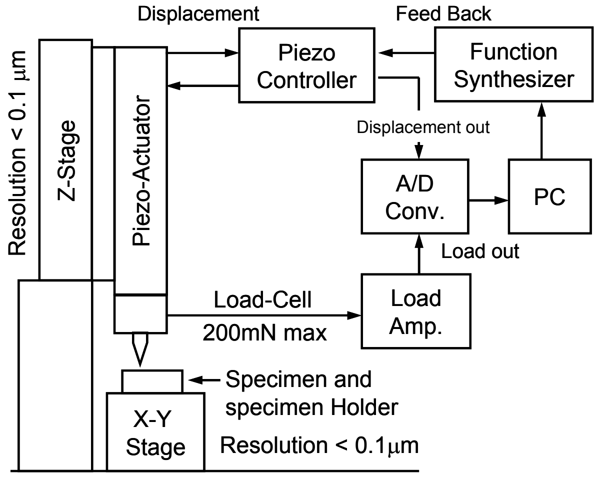

Fig. 3 Block diagram of mechanical testing machine for micro-sized specimens.

3. Results and Discussion

3.1 Fracture Behavior of Millimeter-Sized Specimen

In millimeter-sized specimen, the specimens were fractured brittle manner during introducing fatigue pre-crack as shown in Fig. 4. Figures 5(a) and (b) show scanning electron micrographs of side surface and fracture surface for the millimeter-sized specimen after fracture. The side surface of millimeter-sized specimen after fracture was parallel to the notch plane as shown in Fig. 5(a). The fracture surface was very flat as shown in Fig. 5(b), and the fracture surface was identified to be (100). Therefore, millimeter-sized specimen fractured by cleavage of (100) plane. Fatigue fracture toughness (K_{fc}) was calculated from the maximum cyclic load during introducing the fatigue crack. Stress intensity was calculated by the equation for three point bending specimen [(15)]. K_{fc} of millimeter-sized specimen obtained was 7 MPam$^{1/2}$. This value was very close to fatigue K_{fc} reported for bulk Fe-3 mass%Si alloy single crystal with same orientation [(13)].

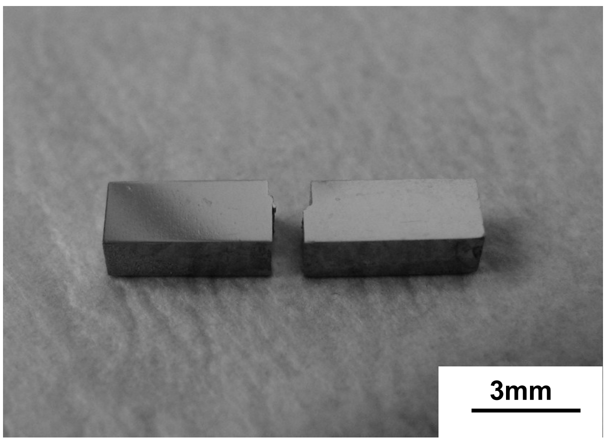

Fig. 4 Photograph of a millimeter-sized specimen after fracture.

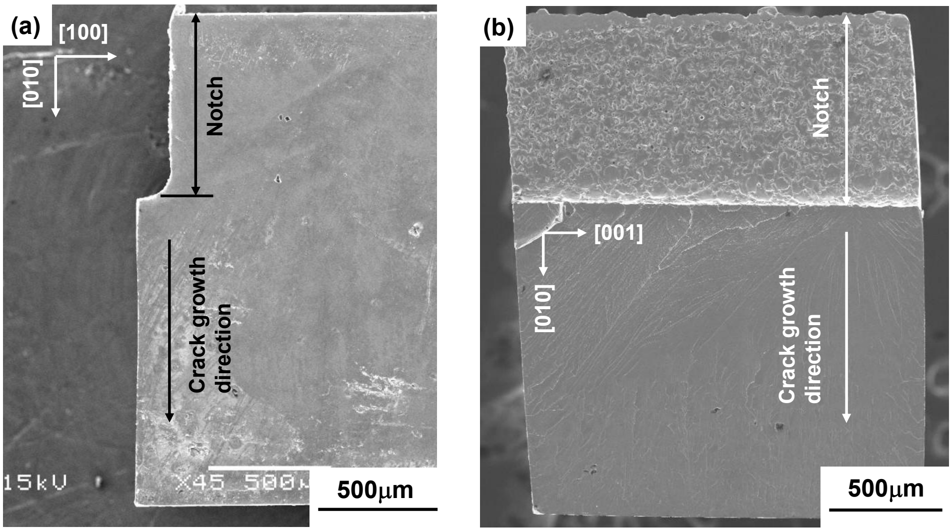

Fig. 5 Scanning electron micrograph of side surface (a) and fracture surface (b) for a millimeter-sized specimen after fracture.

3.2 Fracture Behavior of Micro-Sized Specimen

Figure 6 shows load-displacement curves during fracture testing of micro-sized specimens. In micro-sized specimen, all specimens were showed ductile fracture behavior unlike millimeter-sized specimen. Fracture load values were scattered. Micro-sized specimens were fabricated from thin foils, which were prepared by chemical polishing. The precise control of specimen thickness is difficult in this process. In addition, the control of crack length is also difficult, because the measurement of pre-crack length during application of cyclic loading is impossible for micro-sized specimens. The scatter is, therefore, due to the differences of specimen thickness and the length of fatigue pre-crack.

Figures 7 and 8 show scanning electron micrographs of side surface and fracture surface for micro-sized specimen after fracture tests. Micro-sized specimen fractured by ductile manner, and did not exhibit cleavage fracture unlike the millimeter-sized specimen as shown in Fig. 7.

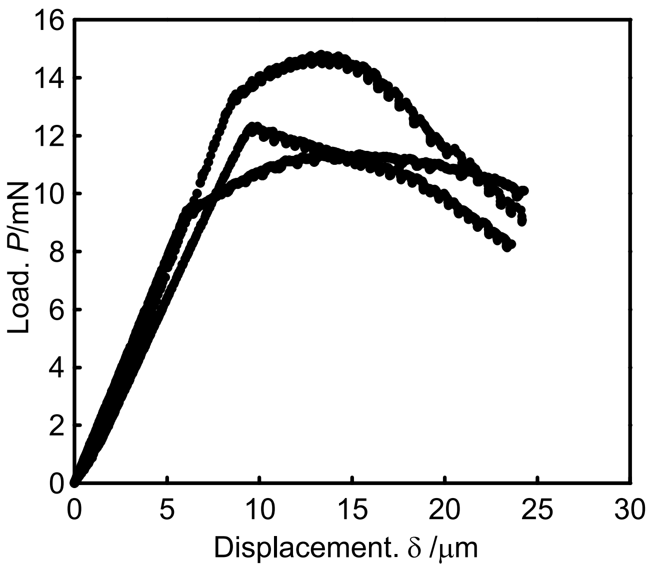

Fig. 6 Load-displacement curves during fracture testing of micro-sized specimens.

Slip lines were observed near the crack tip, and the plastic zone was clearly observed. Furthermore, dimples were also observed on fracture surface as shown in Fig. 8. Thus, in micro-sized specimen, the large scale (general) yielding occurred. As a result of trace analysis of slip lines at the crack tip, slip systems were identified to be $\{11\bar{2}\}<111>$. This slip system was identical with the primary slip system of this bulk material [(13) (16)]. This suggests that the change of fracture mode is not cause by the change of slip systems.

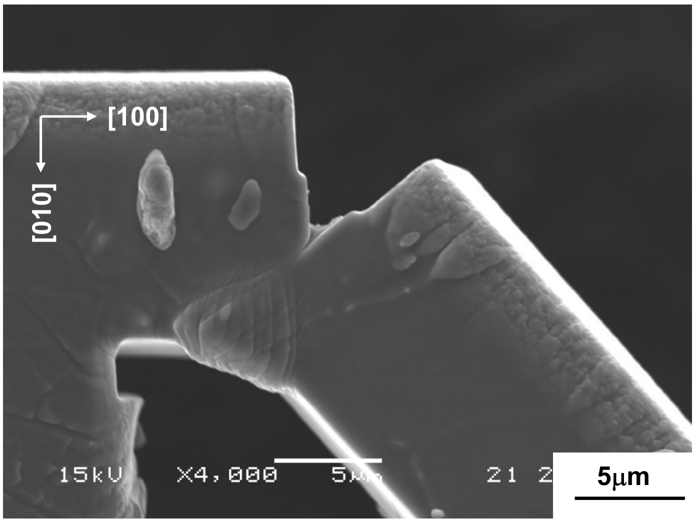

Fig. 7 Scanning electron micrograph of side surface for micro-sized specimen after fracture testing.

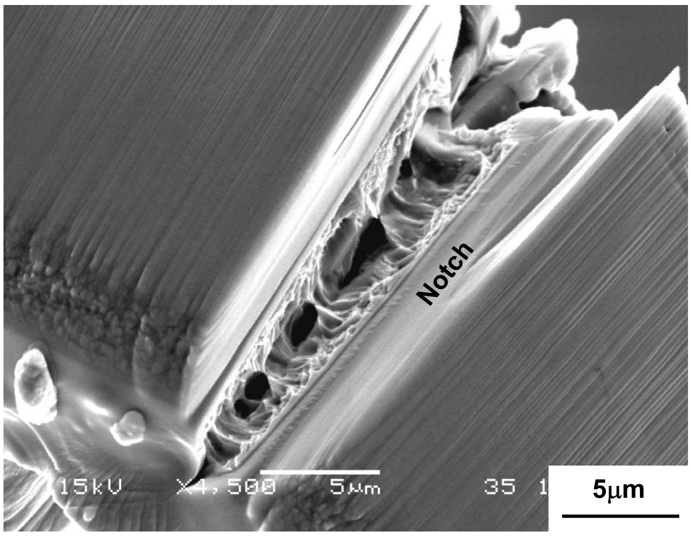

Fig. 8 Scanning electron micrograph of fracture surface for micro-sized specimen after fracture testing.

3.3 Size Effect on Fracture Behavior

As stated above, fracture behavior exhibited the brittle-ductile transition when the specimen size decreases from millimeter- to micro-size. This transition is discussed based on the plastic zone at the crack tip, as the plastic zone size is related to the characteristic length. The plastic zone size at the crack tip under plane stress condition was calculated in the Eq. (1) (15).

$$r_P = \frac{1}{\pi}\left(\frac{K}{\sigma_{ys}}\right)^2 \tag{1}$$

where K is stress intensity factor of the crack tip, σ_{ys} is yield stress of this material. Stress intensity factor is defined by specimen geometry and loading condition, and does not depend on specimen size. In addition, it has been experimentally recognized that σ_{ys} does not depend on specimen size down to micro-size (17). Therefore, the plastic zone size does not depend on specimen size. This indicates that the plastic zone size is the same both for the millimeter-sized and micro-sized specimens. The plastic zone size of this material was calculated to be 90 μm based on the K_{fc} of 7 MPam$^{1/2}$ obtained from the fatigue tests of millimeter-sized specimen and σ_{ys} of 400 MPa of literatures value (18). Figure 9 shows a schematic drawing of the plastic zone size at the crack tip for millimeter-sized and micro-sized specimens. In millimeter-sized specimen, the plastic zone size of 90 μm is satisfied enough small scale yielding condition. In micro-sized specimen, however, this size is much larger than the ligament size and it corresponds to large scale yielding. The size effect on fracture behavior may be related to the plastic zone size at the crack tip and the ligament size of specimen.

The results obtained in this investigation suggest that the size effects on fracture behavior should be considered when micro-sized metallic components were applied for actual MEMS devices. Furthermore, the detail mechanism of the size effects on fracture behavior should be elucidated.

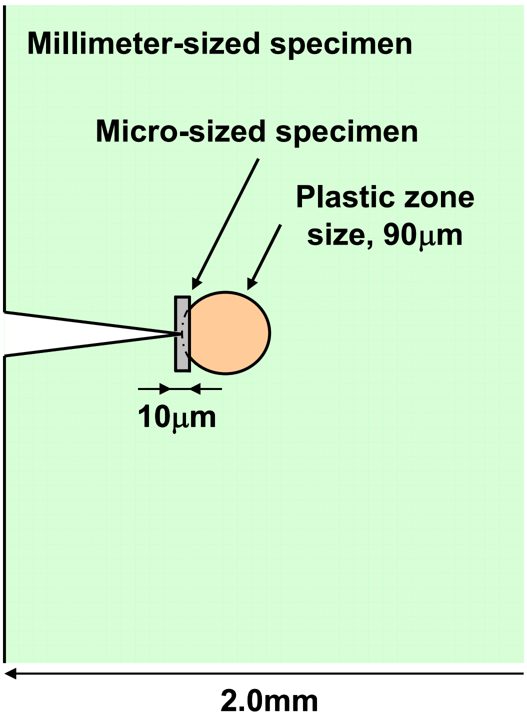

Fig. 9 Schematic drawing of plastic zone size at crack tip for millimeter-sized and micro-sized specimens.

4. Conclusions

Fracture tests have been preformed using both macro-sized and micro-sized specimens prepared from an Fe-3 mass%Si alloy single crystal to investigate the size effect on fracture behavior. The following conclusions were obtained.

(1) Brittle fracture occurred in millimeter-sized specimens, but micro-sized specimens showed ductile fracture and the size effect on fracture behavior was clearly observed.
(2) For millimeter-sized specimen, the plastic zone size is much smaller than the ligament size and small scale yielding occurred. In contrast, micro-sized specimen showed large scale (general) yielding because the plastic zone size is much larger than the ligament size. This may cause the size effect on fracture behavior of this material.
(3) Investigation of size effect on fracture properties of micro-sized materials using MEMS devices was important to ensure reliable and durable of devices.

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